

Esterification of Free Fatty Acids in Crude Palm Oil with Sulfated Zirconia: Effect of Calcination Temperature

Suthat Turapan, Cattareya Yotkamchornkun, and Kamchai Nuithitikul*

Abstract—The production of biodiesel from crude palm oil with a homogeneous base catalyst is unlikely owing to considerable formation of soap. Free fatty acids (FFA) in crude palm oil need to be reduced, e.g. by esterification. This study investigated the activity of sulfated zirconia calcined at various temperatures for esterification of FFA in crude palm oil to biodiesel. It was found that under a proper reaction condition, sulfated zirconia well catalyzes esterification. FFA content can be reduced to an acceptable value for typical biodiesel production with a homogeneous base catalyst. Crystallinity and sulfate attachment of sulfated zirconia depend on calcination temperature during the catalyst preparation. Too low temperature of calcination gives amorphous sulfated zirconia which has low activity for esterification of FFA. In contrast, very high temperature of calcination removes sulfate group, consequently, conversion of FFA is reduced. The appropriate temperature range of calcination is 550-650 °C.

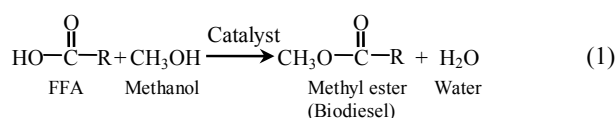
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I. INTRODUCTION

A significant presence of free fatty acids (FFA) in oil used as a raw material for the production of biodiesel can cause several problems. In the typical production of biodiesel with a base homogeneous catalyst (e.g. NaOH, KOH), FFA can react with the base catalyst and consequently generates soap. This soap makes the separation of biodiesel from glycerol more difficult, causing the reduced yield of biodiesel and difficult recovery of glycerol. Moreover the higher amount of FFA in oil, the higher amount of the base catalyst used for the production. This increases the production cost. To avoid these problems, an extra operation or a pre-treatment step is necessary to reduce FFA content in the oil (e.g. to be lower than 1 wt%) before processing it to the typical production of biodiesel with a base catalyst [1-4].

In the pre-treatment step FFA can be esterified with an alcohol, typically methanol, in the presence of an acid catalyst to produce biodiesel as shown in (1). One of very active homogeneous catalysts is sulfuric acid. However it has some drawbacks: corrosive property, difficult separation and higher amount of base needed for neutralizing the remaining acid. To overcome these drawbacks, using an acid heterogeneous

catalyst for esterification of FFA in the pre-treatment step is promising. This can significantly simplify the biodiesel production and reduce the production cost.



Several acid heterogeneous catalysts are reported in the literature [5-9]. Among those, sulfated zirconia ($\text{SO}_4^{2-}/\text{ZrO}_2$) is an interesting one because it possesses strong acidity, referred as superacidity [10-12]. Sulfated zirconia exhibits high catalytic activities in several types of reactions of industrial importance, e.g. acylation [13-14], isomerization [10, 13-14], transesterification [15-16] and esterification [4-5, 15, 17]. However the catalytic activity of sulfated zirconia is enormously varied, depending on the method of preparation.

Sulfated zirconia could be synthesized with several methods, e.g. precipitation [8, 18-19], kneading [15, 17]. The classical preparation method of sulfated zirconia consists of precipitation of zirconium hydroxide from salt solution, impregnation with a sulfating agent, and calcination at high temperatures (e.g. 720 - 970 K) in order to obtain a catalytically active crystalline sulfated zirconia [20]. With this method, types of zirconium precursors and sulfating agents as well as calcination temperature considerably affect the properties and catalytic activity of sulfated zirconia [12].

Although numbers of studies have investigated esterification of a carboxylic substrate with an alcohol [4-5, 15, 17], there has been not any study on esterification of crude palm oil which contains a mixture of FFA and other components. Crude palm oil is a promising and abundant raw material for biodiesel production in Thailand. In this work, therefore, the effect of calcination temperature of sulfated zirconia on its property and catalytic activity for esterification of FFA in crude palm oil to produce biodiesel was investigated. The influence of reaction conditions on esterification of FFA catalyzed by sulfated zirconia was also included.

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II. EXPERIMENTAL

A. Preparation of Sulfated Zirconia

Sulfated zirconia was prepared by the following method [10]. $\text{Zr}(\text{OH})_4$ was prepared by dissolving a required amount of $\text{ZrCl}_2 \cdot 0.8\text{H}_2\text{O}$ (from ACROS) in distilled water and then hydrolyzing at $\text{pH} = 9$ with an aqueous solution of NH_4OH (29.8% from J.T. Baker). The precipitate was stirred, filtered, washed, dried at 110°C for 16 hours, and finally ground. Next sulfated zirconia catalyst was prepared by immersing $\text{Zr}(\text{OH})_4$ in H_2SO_4 (0.5 M), stirring the mixture for 1 hour, drying it at 110°C for 24 hours and calcining the remaining solid at different temperatures for 3 hours. Here we denoted sulfated zirconia calcined at 500, 550, 600, 650 and 700°C as SZ500, SZ550, SZ600, SZ650 and SZ700 respectively.

B. Characterization of Sulfated Zirconia

The specific surface area (S_{BET}) of sulfated zirconia was determined with a Quantachrome Autosorb Automated Gas Sorption System. The BET method with the adsorption data of N_2 at its normal boiling temperature (77 K) was employed. The crystallinity of $\text{SO}_4^{2-}/\text{ZrO}_2$ was determined by powder X-ray diffraction (XRD). The XRD spectra were recorded on a JEOL JDX 3530, using Ni-filtered $\text{Cu K}\alpha$ radiation ($\lambda = 1.540 \text{ \AA}$) at 40 kV, 30 mA. The scanning rate was 2° min^{-1} within the range 2θ of $20\text{--}60^\circ$. Crystallite phases were identified by comparison with the reference data from International Centre for Diffraction Data (ICDD) files. The average crystallite size was calculated from the broadening of the strongest peak using Scherrer equation.

C. Esterification of FFA in Crude Palm Oil

The esterification reaction of FFA in crude palm oil was performed in a stirred-tank reactor equipped with a reflux condenser. 25 mL of crude palm oil (8.8 wt% of FFA) was added into the reactor, followed by a mixture of methanol (8–40 mL) and a prepared sulfated catalyst (0–1.5 g). The reaction temperature was maintained at 60°C . The reaction was initiated by stirring, typically at 250 rpm, and stopped after a specific reaction time for product analysis. The product solution was filtered, washed with water several times and evaporated. The remaining amount of FFA was determined by titration (ASTM D 5555).

III. RESULTS AND DISCUSSION

A. Properties of Sulfated Zirconia

Specific surface area (based on BET model), total pore volume and average pore diameter of sulfated zirconia calcined at various temperatures are reported in Table I. The incorporation of sulfate group onto zirconia significantly increases specific surface area (e.g. from 31.0 to $69.2 \text{ m}^2/\text{g}$ for SZ600) and total pore volume (from 0.13 to $0.19 \text{ cm}^3/\text{g}$), but decreases crystallite size (from 38.5 to 13.1 nm) and average pore diameter (from 16.7 to 11.1 nm).

Based on the greatest values of BET surface area, total pore

TABLE I
PROPERTIES OF SULFATED ZIRCONIA CALCINED AT 500–700 $^\circ\text{C}$

Sample name ^a	S_{BET} (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (nm)	Crystallite size ^b (nm)
SZ500	77.2	0.09	4.7	–
SZ550	95.1	0.13	5.5	10.1
SZ600	69.2	0.19	11.1	13.1
SZ650	72.0	0.14	7.9	12.6
SZ700	65.8	0.11	6.5	15.1
Z600	31.0	0.13	16.7	38.5

^aSZ500 = sulfated zirconia calcined at 500°C ; SZ550 = sulfated zirconia calcined at 550°C ; SZ600 = sulfated zirconia calcined at 600°C ; SZ650 = sulfated zirconia calcined at 650°C ; SZ700 = sulfated zirconia calcined at 700°C ; Z600 = zirconia calcined at 600°C .

^bDetermined from XRD patterns using Scherrer equation at $2\theta = 30^\circ$

volume and average pore diameter, there are different optimum calcination temperatures. The greatest specific surface area was obtained with sulfated zirconia calcined at 550°C whereas the greatest total pore volume and biggest pore diameter were found with sulfated zirconia calcined at 600°C . Referred to the average pore diameters in Table I, the synthesized sulfated zirconia in this study has mesoporous structure. The mesoporous catalysts are primarily preferred for biodiesel production which usually deals with large reactant molecules having long-chain alkyl group [21].

XRD patterns of sulfated zirconia calcined at 500–700 $^\circ\text{C}$ are shown in Fig.1.

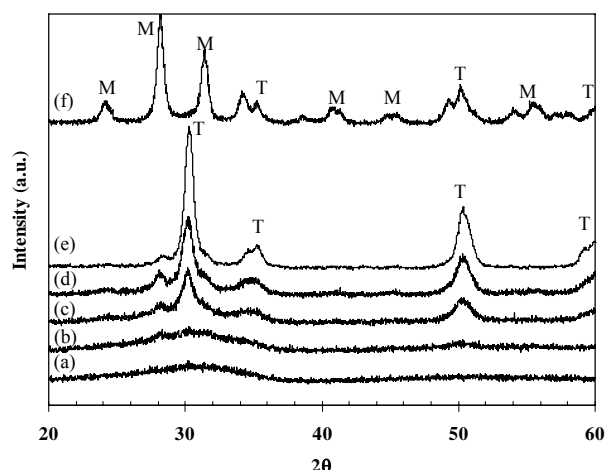


Fig. 1 XRD pattern of sulfated zirconia calcined at (a) 500°C ; (b) 550°C ; (c) 600°C ; (d) 650°C ; (e) 700°C ; (f) zirconia calcined at 600°C

Incorporation of sulfate onto zirconia at 600°C changes its crystallite structure from the mixture of monoclinic (M) and tetragonal (T) phases to only the tetragonal phase at 2θ of 30° , 35° , 50° and 60° . The crystallite size is significantly decreased with the sulfate attachment. These correspond to the major part of literature results [12, 20, 22, 23].

Increasing calcination temperature of sulfated zirconia from 500°C to 700°C increases crystallinity. Calcining sulfated zirconia at low temperature, i.e. 500°C , gives amorphous structure. This corresponds to a previous research that found

amorphous structure when calcining sulfated zirconia at 400 °C [5]. Calcining sulfated zirconia at 550-700 °C increases the crystallite size as indicated by the increased peaks' intensity with temperature. Only tetragonal phase is formed when calcining sulfated zirconia at 550-700 °C, which corresponds to [24].

B. Effect of Calcination Temperature

The effect of calcination temperature on the activities of sulfated zirconia calcined at 500-700 °C is shown in Fig. 2.

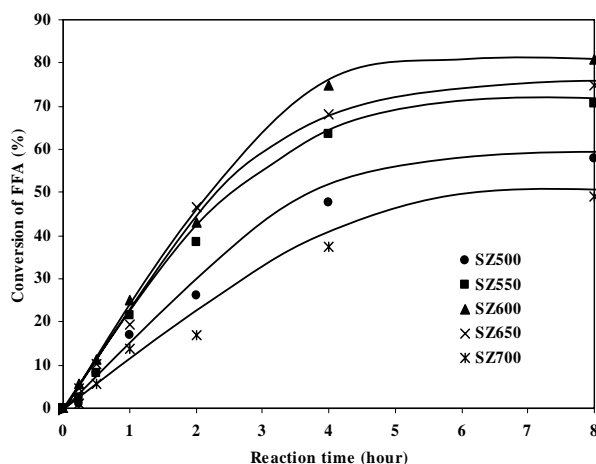


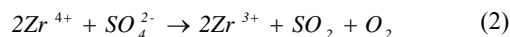
Fig. 2 Effect of calcination temperature of sulfated zirconia on conversion of FFA [reaction conditions: 0.5 g of catalyst, 30 mL of methanol]

As shown in Fig. 2, sulfated zirconia calcined at 600 °C exhibits the highest activity. At 4 and 8 hours of reaction, the highest conversions of FFA are obtained: 75.0% and 80.7% respectively. The highest activity of SZ600 corresponds to its highest pore diameter (11.1 nm) and pore volume (0.19 cm³/g) as indicated in Table I. Since FFA molecules are large and consist of long alkyl chains, pore diffusion, rather than specific surface area, becomes an important factor. The pore diffusion limitation can be minimized when a catalyst with larger pore size is employed.

However the initial reaction rates (determined from the initial slopes) with sulfated zirconia calcined at 550, 600 and 650 °C are not much different; they are 8.5×10^{-7} , 9.9×10^{-7} , and 9.3×10^{-7} mol/g.s respectively. For sulfated zirconia calcined at 500 and 700 °C, the initial reaction rates are 5.0×10^{-7} and 4.3×10^{-7} mol/g.s respectively, which are significantly lower than those obtained from sulfated zirconia calcined at 550-650 °C. Therefore it can be said that the temperature range of 550-650 °C is appropriate for calcining sulfated zirconia to be an active catalyst for esterification of FFA in crude palm oil. It is noted that the optimum calcination temperature of sulfated zirconia is varied, depending on the reaction system, e.g. 525 °C for esterification of acetic acid with butanol [5], 575 °C for esterification of acetic acid with ethanol [25], 620 °C for esterification of benzoic acid with methanol [26].

Calcining sulfated zirconia at very low temperature (e.g.

<550 °C) and very high temperature (e.g. >650 °C) decreases the activity of sulfated zirconia. Calcining sulfated zirconia at too low temperature does not create the crystallinity of sulfated zirconia. Amorphous structure is eventually obtained. In contrast, calcining sulfated zirconia at very high temperature promotes tetragonal structure but loosens sulfate groups from the structure. Some sulfates can decompose to form SO₂ at high temperature as shown in (2) [12]. Since sulfates are the active sites for esterification, losing them means the loss of catalyst activity. As a result, the conversion of FFA catalyzed by SZ700 is the lowest.



Reference [4] has found that sulfated zirconia catalyst calcined at temperature below 400 °C was essentially not active whereas calcining sulfated zirconia at 500 and 600 °C gave the catalyst with higher and similar activities, i.e. 35-45% conversion of palmitic acid at 60 °C and 2 hours of reaction. This is in agreement with our study; sulfated zirconia calcined at 500 and 600 °C gave 26.1% and 43.2% conversions of FFA. However conversions of FFA in our study are lower due to the lower BET surface area of the catalyst. Sulfated zirconia calcined at 600 °C in reference [4] had higher BET surface area (103 m²/g) and smaller average pore diameter (1.17 nm) than ours (see Table I).

With sulfated zirconia calcined at 600 °C, the FFA content of the crude palm oil could be reduced from 8.8 wt% to 1.7 wt% (80.7% conversion) within 8 hours of the reaction. This palm oil is, therefore, able to be used further for the typical biodiesel production with a base homogeneous catalyst (e.g. NaOH) to prevent significant formation of soap and yield loss of biodiesel. However 8 hours of reaction time are still too long. Increasing the reaction temperature to be higher than 60 °C is recommended for enhancing the reaction rate. In esterification of n-octanoic acid with methanol at 175 °C, sulfated zirconia-alumina catalyst gave 99% conversion after 20 hours of reaction [15]. However its activity dropped significantly when the reaction temperature was below 120 °C [17].

As shown in Fig. 2, the conversion of FFA considerably increases with reaction time during the first 4 hours and then a gradual increase is established. It is likely that the reaction equilibrium is achieved before 100% conversion of FFA. Esterification is typically a reversible reaction. When the reaction takes place, water product is generated and this will inhibit the forward reaction of FFA. In our study on the effect of water content, higher amount of water in the reaction mixture actually reduces the conversion of FFA.

Paying attention to the highest conversion of FFA with sulfated zirconia calcined at 600 °C, further studies in the effect of amount of methanol, amount of sulfated zirconia, and water content were based on SZ600.

C. Effect of Amount of Methanol

Variation in ratio of methanol to crude palm oil affects the

conversion of FFA as shown in Fig. 3. The conversion of FFA proportionally increases to the amount of methanol up to 30 mL. Further increase in the amount of methanol slightly enhances the conversion of FFA. For a reversible reaction like esterification, excess amount of methanol is typically required to push the reaction forward the formation of products. However too much methanol could dilute the system and eventually cause slower reaction [21]. Excess amount of methanol might saturate the catalytic surface with the methanol or prevent nucleophilic attack by shielding the protonated methanol by its own excess [27].

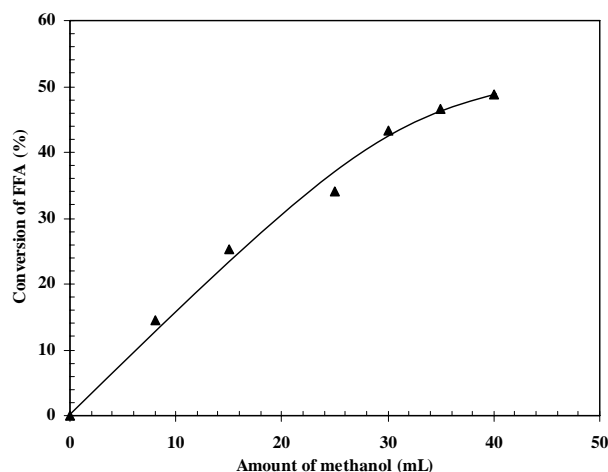


Fig. 3 Effect of amount of methanol on conversion of FFA [reaction conditions: 0.5 g of SZ600, 2 hours of reaction]

D. Effect of Amount of Sulfate Zirconia

When the amount of sulfated zirconia calcined at 600 °C is increased, the conversion of FFA in crude palm oil increases and begins to level off later as shown in Fig. 4. This indicates mass transfer limitation at high catalyst loading. Although more available active sites are increased with catalyst loading, overall reaction rate cannot be enhanced if the diffusion of the reactants to the active sites occurs slowly. According to Fig. 4, the optimum amount of sulfated zirconia to catalyze the esterification reaction at 60 °C is 1 g.

It is important to note that esterification of FFA in crude palm oil can occur even in the absence of the catalyst; the conversion of FFA was 8.0 %. This is similar to a previous study [28] who found that oleic acid in soybean oil could be reduced from 10 wt% to about 6.4 wt% by esterification with methanol at 180 °C for 1 hour without any catalyst addition.

A. Effect of Water Content

The original water content in crude palm oil and methanol was 0.25 and 5.0 wt% respectively. Based on the amount of oil (25 mL) and methanol (30 mL), the total water content in the reaction mixture was 2.7 wt%. To investigate the effect of water content, we added extra amount of deionized water into the reaction mixture before initiating the reaction. The total water contents were, therefore, 3.5, 6.0 and 9.0 wt%. As

shown in Fig. 5, water content in the reaction mixture negatively affects esterification of FFA. Moreover conversions of FFA catalyzed by both SZ500 and SZ600 decrease with similar degree.

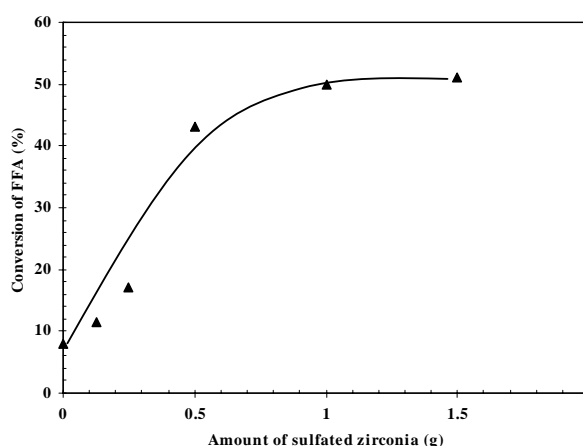


Fig. 4 Effect of amount of sulfated zirconia (SZ600) on conversion of FFA [reaction conditions: 30 mL of methanol, 2 hours of reaction]

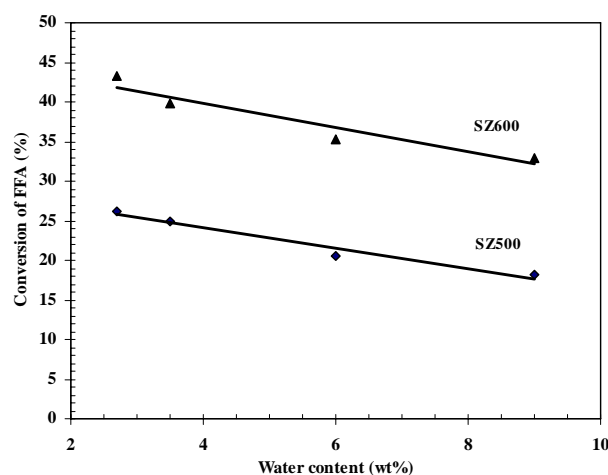


Fig. 5 Effect of water content on conversion of FFA [reaction conditions: 0.5 g of SZ600 or SZ500, 30 mL of methanol, 2 hours of reaction]

The decrease in conversion of FFA with the addition of water corresponds to a previous study [3] who found that addition of water (3800 ppm) significantly reduced conversion of palmitic acid in soybean oil reacting with methanol at 85 °C, using multifunctionalized mesoporous silica as a catalyst. This is due to that the presence of water inhibits the forward reaction. Moreover the presence of water might have a negative effect on the activity of sulfated zirconia as pointed out by reference [4] that contacting sulfated zirconia with water almost fully inhibited its activity for esterification of palmitic acid in sunflower oil with methanol.

IV. CONCLUSION

Sulfated zirconia has been proved to be a promising heterogeneous catalyst for the esterification of FFA in crude palm oil. It can significantly reduce the FFA content to be lower than 1 wt% (an acceptable value for oil feedstock used in the production of biodiesel with a homogeneous base catalyst) when an appropriate reaction condition is employed, e.g. at a reaction temperature higher than 60 °C and with sulfated zirconia calcined at 550-650 °C. Further research should be carried out in terms of the effect of sulfate content in zirconia, type of sulfating agents and, in particular, the catalyst activity after several uses.

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